

Evaluation of Parameters Affecting
Activated Carbon Adsorption of a
Solvent-Laden Air Stream

IT Corp., Knoxville, TN

Prepared for

Environmental Protection Agency, Cincinnati, OH

Jul 85



U.S. Department of Commerce
National Technical Information Service

NTIS

EPA/600/D-85/144
July 1985

EVALUATION OF PARAMETERS AFFECTING ACTIVATED
CARBON ADSORPTION OF A SOLVENT-LADEN AIR STREAM

By

Michael L. Foster
IT Corporation
312 Directors Drive
Knoxville, Tennessee 37923

Ronald J. Turner
U.S. Environmental Protection Agency
Hazardous Waste Engineering Research Laboratory
Cincinnati, Ohio 45268

HAZARDOUS WASTE ENGINEERING RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OH 45268

TECHNICAL REPORT DATA (Please read Instructions on the reverse before completing)		
1. REPORT NO. EPA/600/D-85/144	2.	3. RECIPIENT'S ACCESSION NO. PBB 5 235992/AS
4. TITLE AND SUBTITLE Evaluation of Parameters Affecting Activated Carbon Adsorption of a Solvent-Laden Air Stream		5. REPORT DATE July 1985
		6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) Michael L. Foster		8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS IT Corporation 312 Directors Drive Knoxville, Tennessee 37923		10. PROGRAM ELEMENT NO. C9HA1A
		11. CONTRACT/GRANT NO. 68-02-3917
12. SPONSORING AGENCY NAME AND ADDRESS Hazardous Waste Engineering Research Laboratory Office of Research and Development U.S. Environmental Protection Agency Cincinnati, OH 45268		13. TYPE OF REPORT AND PERIOD COVERED Conference Paper
		14. SPONSORING AGENCY CODE EPA/600/12
15. SUPPLEMENTARY NOTES		
16. ABSTRACT As part of EPA's continuing research on methods to control volatile organic compound emissions, a vapor-phase activated carbon pilot plant has been constructed. This automated, computer-controlled pilot plant has been designed to provide data not easily obtained from full-scale adsorption systems. As part of an overall testing program, the pilot plant was operated on an actual solvent-laden air stream at a manufacturing site. The purpose of the testing was to evaluate parameters affecting vapor-phase carbon adsorption and to determine if modifications to the existing adsorption system at this site would improve performance. The parameters investigated include relative humidity and temperature of the solvent-laden air stream, carbon type, the amount of steam used to regenerate the carbon, and the effect of cooling and drying the carbon bed after regeneration. The experimental conditions and the loading, expressed in pounds of volatile organic compound adsorbed per pound of activated carbon, are reported.		
17. KEY WORDS AND DOCUMENT ANALYSIS		
a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
VOC Reduction by Carbon Adsorption	Carbon Adsorption VOC Control Solvent Recovery	13B
18. DISTRIBUTION STATEMENT Release	19. SECURITY CLASS (This Report) Unclassified	21. NO. OF PAGES 17
	20. SECURITY CLASS (This page) Unclassified	22. PRICE \$700

NOTICE

This document has been reviewed in accordance with U.S. Environmental Protection Agency policy and approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

INTRODUCTION

Vapor-phase carbon adsorption is widely used in various industries for controlling emissions and for recovery of valuable solvents. Previous projects funded by the EPA have collected performance data on existing full-scale adsorption systems. However, due to the constraints involved in full-scale systems, variation of parameters for the purpose of studying adsorption effectiveness under a variety of conditions was impractical. The process from which a solvent-laden air (SLA) stream is created, the compounds involved, and the climate can all impact the carbon adsorption process. Therefore, a pilot-scale unit was needed which could be easily manipulated to operate under a wide range of conditions. The pilot plant was to be of such a scale that the conclusions drawn from its operation would be applicable to full-scale systems.

With the foregoing in mind, the goals of the project were developed. The initial task was to design and construct a pilot-scale adsorption system with the desired versatility. The following were considered to be the adsorption variables: carbon type, relative humidity, solvent-laden air temperature, concentration of volatile organic compound (VOC) and VOC type, steam regeneration ratio, and the cooling and drying step. In order for the pilot plant to be useful, it also had to be reasonably transportable so that it could be moved to production facilities and perform tests on actual process streams. Other design constraints included automating and computerizing the system operation to minimize both the operator time and bias. Also, the adsorption equipment had to be explosion proof so that it could be placed in high hazard areas of production facilities.

Following procurement, construction, and shakedown a series of tests were run by using a spraying system to generate a simulated SLA stream. The conditions of adsorption were varied to determine the effects on loading of VOC on the carbon bed. Once this series of tests was completed, the pilot plant was moved to two field sites in order to gain information on actual process streams containing volatile organic compounds.

This paper describes the design and operation of the EPA carbon adsorption pilot plant and presents the results of tests performed at one of the field sites.

DESCRIPTION OF PILOT PLANT

Equipment

The pilot plant equipment is divided onto four skid-mounted units plus a computer and an analyzer. The four skids are the adsorption skid, blower skid, chiller skid, and boiler skid.

The adsorption skid is the heart of the pilot plant. On this skid are two identical stainless steel adsorbers. The adsorbers are 2 feet in diameter and can accommodate bed depths up to 2 feet. This skid also contains the heat exchangers, filters, dilution system, and flow control system necessary to condition the solvent-laden air and to permit adsorption under controlled parameters. Numerous sensors and power-actuated valves provide the capability to cycle automatically through various modes of operation. Finally, there is a condenser and phase-separation system for recovery of solvent.

The blower skid is connected to the adsorption skid to provide the draft necessary to move the SLA through the adsorbers. The blower is a multi-stage centrifugal type, driven by a 7.5 horsepower electric motor.

The chiller skid provides chilled coolant to the adsorption skid for conditioning incoming SLA and for condensing regenerated solvent vapors. The chiller is a water-cooled, 20-ton unit, with a centrifugal pump on this skid to circulate the coolant.

Steam for regeneration of loaded carbon beds can be provided from either an external source or by the boiler skid. The steam generator is capable of delivering 200 pounds per hour of low-pressure steam.

The Byron Model 401 total organic analyzer provides analyses of the air entering and exiting the carbon adsorbers. The results are used to plot breakthrough curves and to determine loadings of the VOC on the carbon. A Byron Model 10-2 sample pump continuously draws the samples from the adsorption skid and through the analyzer.

The computer, used for data logging and process control, is a standard IBM personal computer. The computer is equipped with 256 K bytes of RAM, two double-sided, double-density disk drives, and a monochrome screen. There is also a 80 CPS parallel printer and a modem. The accompanying software allows the operator to implement a variety of control strategies through a system of easily configured building blocks.

Three main modes of operation may occur during each adsorption cycle. In the adsorption mode, SLA is passed through a bed of activated carbon. When the carbon bed becomes loaded to the degree that the effluent concentration rises beyond a predetermined level, breakthrough has occurred and the carbon must be regenerated. Regeneration is accomplished using low pressure steam. Following regeneration a cooling and drying step is normally used to return the carbon bed to a more suitable condition for adsorption. The following is a more detailed description of how each mode operates. Two other modes of operation, which exist for safety reasons, are the deluge mode and the bypass mode.

Adsorption Mode

In this mode of operation, gas containing a volatile organic compound is passed through one of the two identical carbon beds to remove the VOC. Figure 1 is a diagram of the pilot plant with the emphasized line showing the path of flow during the adsorption mode.

The SLA is drawn in through a flow control valve, filtered to remove particulate and then passed through a series of two heat exchangers and a cyclone. The purpose of the heat exchangers and cyclone is to adjust the temperature and relative humidity of the SLA.

The first heat exchanger uses chilled water to bring the temperature of the SLA below its dewpoint. The exact temperature is determined by locating on a psychrometric chart the point which represents the desired dry bulb temperature and the desired relative humidity. The corresponding saturation temperature may then be read and this temperature is used as the set point for the exchanger. If the incoming SLA does not contain sufficient moisture to be saturated at the chilled water exchanger set point, then steam is injected prior to the chilled water exchanger.

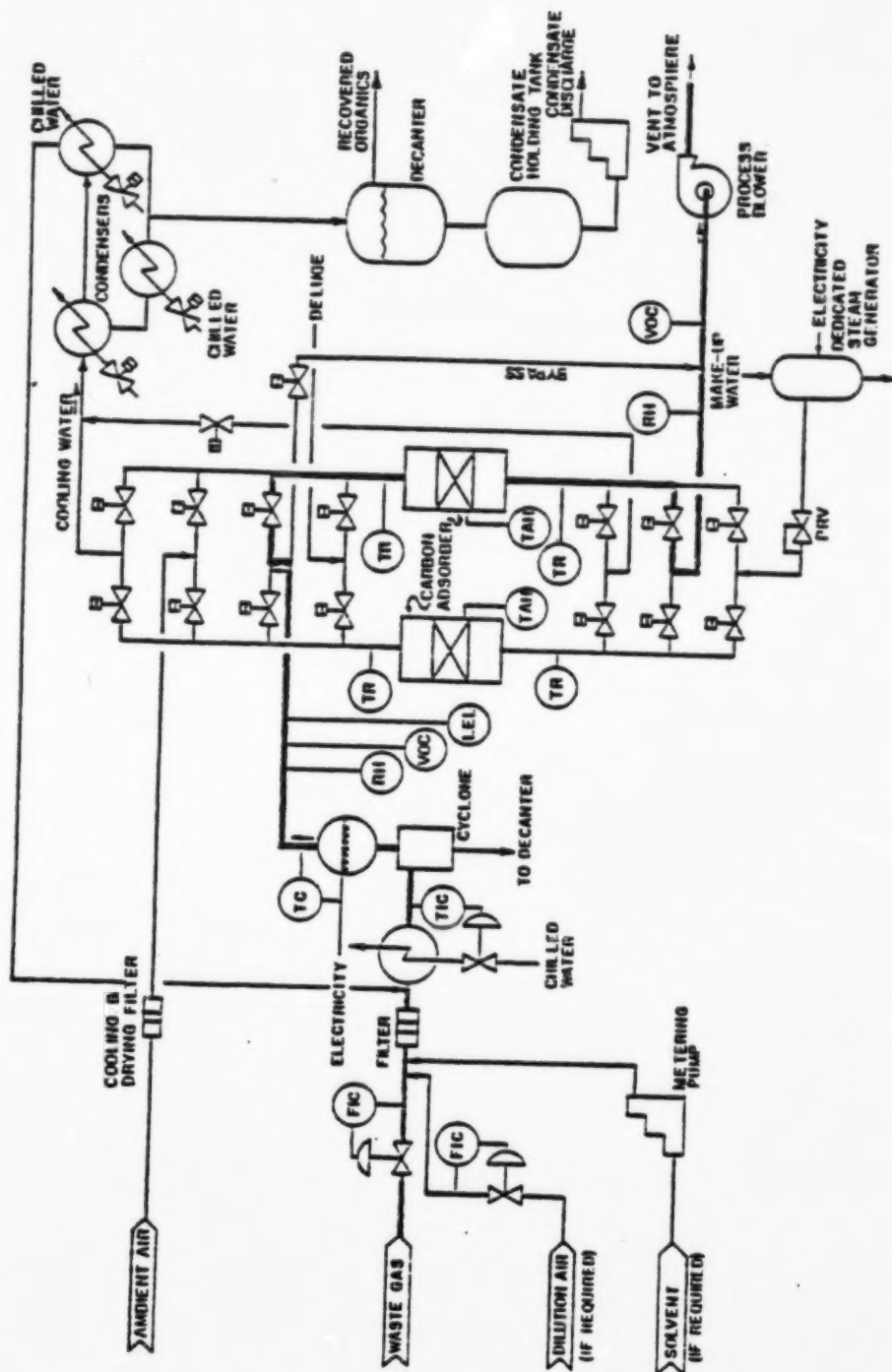


FIGURE 1. PATI OF SLA FLOW

After the chilled water exchanger, condensed water is removed in a cyclone. The cyclone is necessary to achieve effective separation of the entrained water. The SLA is then heated to the desired dry bulb temperature by an electric heater. The SLA is drawn through the adsorber and exhausted by the process blower. The total pressure drop in the system is approximately 80 inches of water. Adsorption takes place under a vacuum of about 70 inches of water.

If the SLA must be generated or if the concentration of the VOC in the SLA needs to be increased, a solvent spraying system can be used. The sprayer uses compressed air to atomize the solvent and assure complete evaporation and mixing of the solvent in the air stream before reaching the carbon bed. A metering pump is used to control the solvent flow.

Regeneration Mode

Regeneration of the loaded carbon beds is accomplished by steaming in the reverse direction from adsorption. The path of the regeneration steam is shown in Figure 2. The steam containing VOC vapor travels overhead into a condenser operating on 70°F cooling water. Noncondensibles pass to a secondary chilled-water condenser. Both of these condensers drain into a decanter. When a layer of organic is formed, it is decanted continuously from the aqueous condensate.

Cooling and Drying Mode

At the end of the regeneration process the carbon bed is hot and full of steam and/or condensate. An optional cooling and drying step may be used to bring the carbon bed to conditions more suitable for adsorption. The path of the cooling and drying flow is shown by the emphasized line in Figure 3.

Filtered ambient air is used for this purpose. The air passes through the adsorbers and through the same heat exchangers as the steam did in the regeneration step. The cooling and drying air is recycled to the incoming SLA to prevent loss of solvent.

Bypass Mode

There are two situations that can cause the SLA flow to be routed around the adsorbers and exhausted. They are a high temperature condition in a carbon bed and a high LEL reading of the SLA stream. Whenever either of these conditions exists, appropriate valves close to stop adsorption and a bypass valve opens. In the case of high LEL, a return to normal readings will allow adsorption to resume. Normally, the LEL trip is set at 25 percent of the LEL.

Deluge Mode

The deluge mode is intended to protect both the pilot plant and surrounding area in the event a fire should occur in the carbon beds. When a temperature is detected in either bed which exceeds 250°F, both beds are isolated, the bypass opens, and a deluge is initiated. The water deluges only the bed where the high temperature occurred and ceases as soon as the temperature drops below 250°F. However, once initiated, the position of the valves can no longer be changed automatically by the computer. This is to prevent an adsorption mode from being restarted after a bed has been flooded.

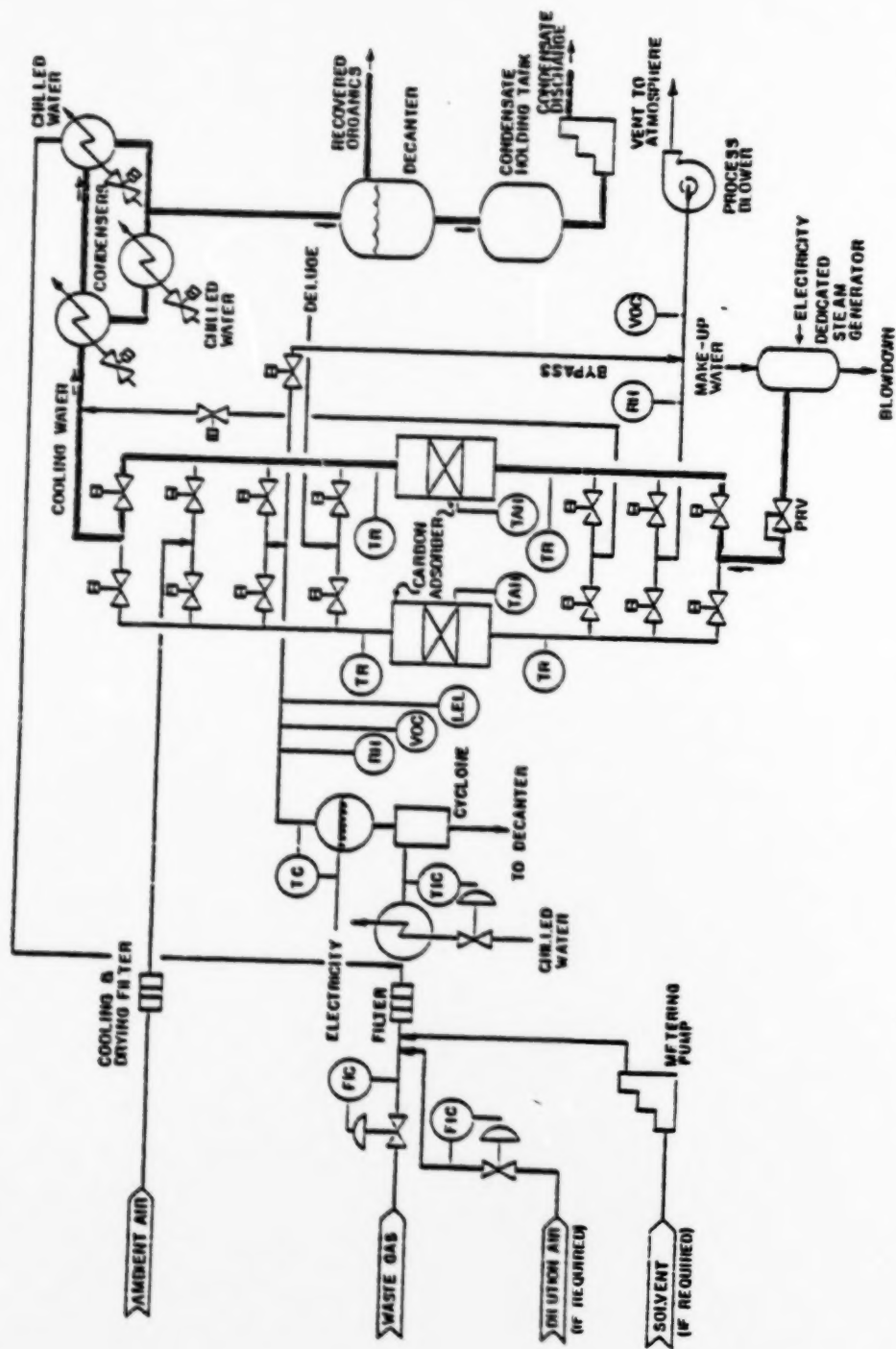


FIGURE 2. PATH OF REGENERATION FLOW

9350.05/SCK-WTS/M-26-84

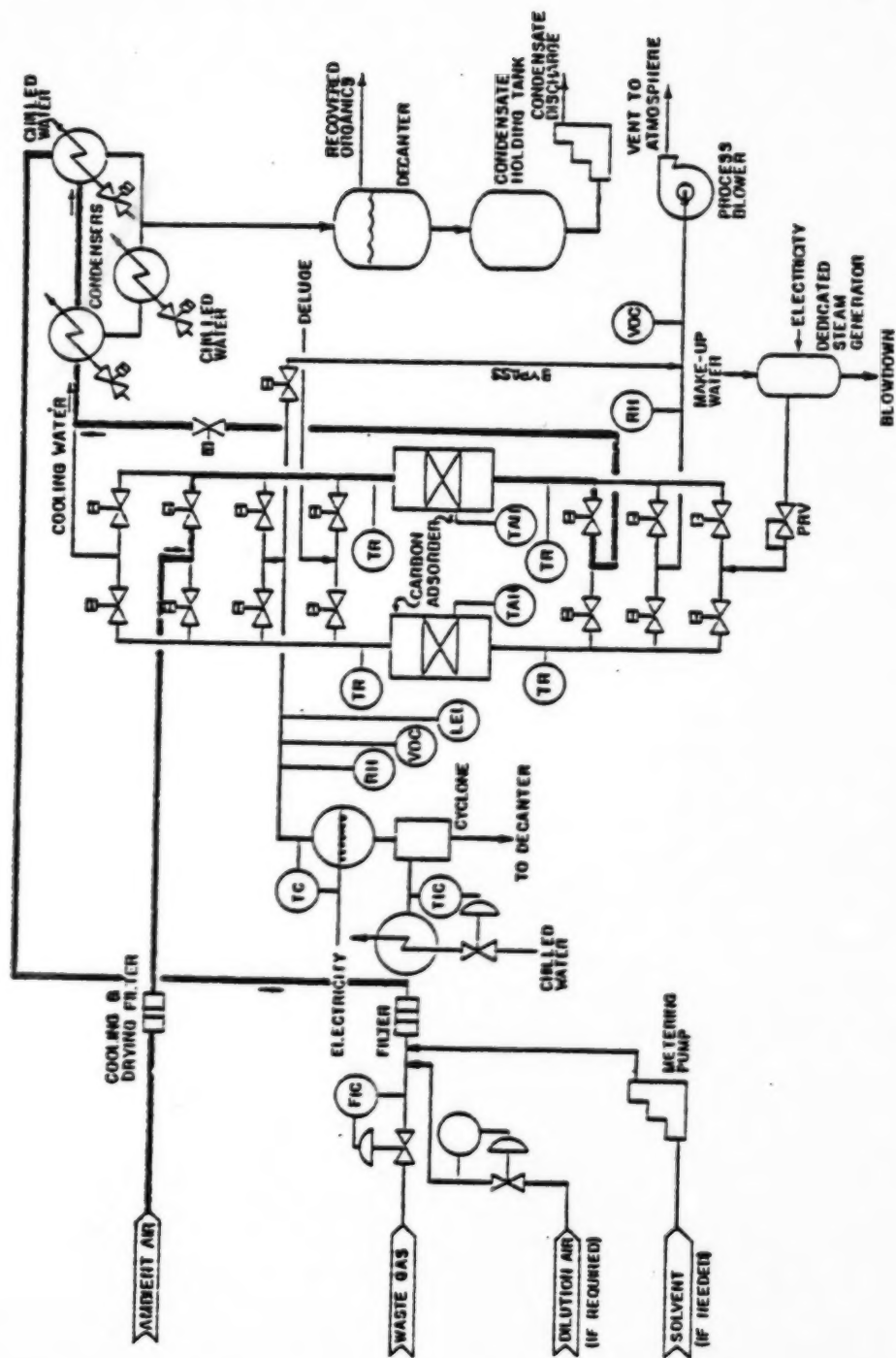


FIGURE 3. PATH OF COOLING AND DRYING FLOW

0350.05/5CK-WTS/10-26-84

EXPERIMENTAL APPROACH

Data Generation

The data, including all temperatures, flows, LEL readings, and the inlet and outlet VOC concentrations, were recorded automatically by the IBM PC. Data were usually recorded at 6 minute intervals in order to match the regular output of the Byron 401 analyzer. Some data such as pressure drops through equipment were recorded on a random basis from local indicators. Data were printed on paper and stored on a computer floppy disk in order to facilitate subsequent data analysis and virtually eliminate the errors that result from key punching.

The most critical data source on this project was the Byron 401 analyzer. The analyzer contains separate channels for measurement of total hydrocarbons, non-methane hydrocarbons, methane, carbon dioxide, and carbon monoxide. Only the non-methane hydrocarbon channel was used during this project.

In the Byron 401 analyzer, the non-methane hydrocarbons are chromatographically isolated from methane, carbon monoxide, and carbon dioxide. The non-methane hydrocarbons are then completely oxidized into carbon dioxide and completely reduced to methane before being measured in a hydrogen flame ionization detector. Response factors for different compounds have been eliminated because the detector measures only methane. This allows the use of reliable propane standards to calibrate the instrument for any compound desired.

Data Reduction

The data were reduced by transferring into a spread sheet program where calculations were made on the critical operating parameters. For example, the temperature of the inlet SLA was averaged and the duration of the breakthrough curve was calculated.

Evaluation of the performance of the carbon under different conditions is based on the pounds of VOC that were adsorbed on a pound of carbon. Adsorption loadings were calculated using the temperature corrected flow and the inlet and outlet concentrations over each data logging interval. Calculations were performed for each data interval from the beginning of adsorption until the point where the effluent concentration reached breakthrough. The results for each interval were summed to get the overall loading. The following equation shows how the loadings were calculated for each data interval.

$$IL = (VOC * F * T * C) / (1 * 10^6 * W)$$

where:

- VOC = feed concentration-effluent concentration (ppm v/v);
- F = flow of SLA at 70°F and at 1 atm (cubic feet/minute);
- T = data interval (minutes);
- W = weight of carbon in bed (pounds);
- C = conversion from cubic feet of VOC to pounds:
(1 pound mole/387 cubic feet)* (92.1 pounds toluene/pound mole toluene); and
- IL = interval loading (pounds VOC/pound carbon).

Breakthrough on this project was set at an effluent concentration of 10 percent of feed. Since the majority of the runs were conducted with target inlet VOC

concentrations of 2000 ppm v/v, the breakthrough point was 200 ppm v/v outlet concentration. Set point for the flow was 150 cfm, and the data interval was either 3 or 6 minutes. A normal carbon charge in the adsorbers weighed about 200 pounds.

FIELD TESTING

Process Description

Following completion of a shakedown and matrix testing phase, the VOC pilot plant was moved to an actual production facility for further testing. Pressure-sensitive tape is made at this facility. Toluene, used as a solvent for the adhesive applied to the tape, is vaporized in ovens and drawn to a solvent recovery system. The solvent-laden air also contains some oil and heavy polymers from the process.

The solvent recovery system is composed of activated carbon columns which are steam regenerated. The main purpose of the system at this site is recovery and reuse of the toluene. Normal recovery efficiencies are 90 percent or better. The company agreed to participate in this project in order to fine-tune its adsorption system for maximum efficiency.

A slip stream from the main solvent-laden air duct was used as supply for the pilot plant. Because of the fact that this is a solvent recovery system on a real process, the concentration of toluene was not always constant. The concentration varied depending on the type and amount of product being produced. The concentration was also affected by production lines going down. This was especially noticeable if only one or two lines were running. Concentration therefore was not considered to be a controlled variable in these runs. Generally, under normal conditions the concentration of the toluene was 2000 plus or minus 500 ppm v/v.

In Table 1 the target conditions for the field site testing are shown.

Table 1. Target Conditions for Field Site Testing

Test No.	VOC	SLA Inlet Temperature (F)	Relative Humidity (%)	Regeneration Steam (lbs/lb carbon)	Cooling/Drying (minutes)	VOC Inlet Concentration (ppm v/v)
1	Toluene	90	30	0.5	60	2000
2	Toluene	90	60	0.5	60	2000
3	Toluene	90	90	0.5	60	2000
4	Toluene	90	60	0.5	0	2000
5	Toluene	90	60	0.3	60	2000
6	Toluene	70	90	0.5	60	2000
7	Toluene	110	60	0.5	60	2000

RESULTS

Initial cycles of any particular test will be affected by the prior history of the carbon. If all data for every cycle of every test were plotted, the results would be less likely to be representative of the actual conditions and the trends would be less obvious. Therefore, only the results from the last 50 percent of any test have been plotted. If an odd number of cycles were completed during a test, then the remaining extra cycle was plotted. For example, if 8 cycles were completed on a carbon bed at a given set of conditions then only the last 4 were plotted. If only three cycles were completed, then only the last two were plotted. In no case was the first cycle at any condition plotted because it was felt that the first cycle result would represent transition from one set of conditions to the next set.

During the entire testing program at this field site, activated carbons derived from two distinctly different sources were used. The first, Witcarb Columbia JXC, is made from petroleum pitch. The second, Cecarbon Acticarbone AC 35, is made from pinewood charcoal. These carbons were chosen because they are widely used in industrial adsorption systems. The carbons were placed in the identical adsorbers and tested side-by-side to minimize differences in test conditions.

The following text is divided into a discussion of the effects of each parameter on the loading of the VOC. Enough data were collected on the following parameters to allow the creation of graphs: relative humidity, temperature, cooling and drying step, and steam regeneration ratio. In addition, the results from the two activated carbons are represented by the different data point symbols.

Each graph shows the effect of the particular parameter when the other target conditions were being held constant. Generally, the actual conditions during a test were reasonably close to the target conditions. Tables of the average test conditions are available from the author.

Relative humidity was tested at 30, 60 and 90 percent. Figure 4 shows the effect of increasing relative humidity on the loading of the solvent. A reduction in loading of about 50 percent can be expected when relative humidity increases from 30 percent to 90 percent.

Temperatures of 70, 90 and 110 degrees Fahrenheit were tested. Two different relative humidity levels were involved as shown in Figure 5.

The regeneration steam ratio is defined as the pounds of steam used during regeneration divided by the pounds of carbon in the bed. At this field site ratios were varied from 0.3 to 0.5 pound steam/pound carbon. Figure 6 shows the effect of the change in the regeneration steam ratio. The steam flow at this site was difficult to control. The line pressure varied depending on process demands such as whether the full scale adsorption towers were being regenerated. The steam supply pressure was several hundred pounds and had to be reduced by a pressure regulator.

Normally it is desirable to have a cooling and drying cycle between the regeneration cycle and the adsorption cycle. This step partially removes the heat and moisture from the carbon leaving it in a state more suitable to resume adsorption. The goal was to test the effect that cooling and drying had on the subsequent loading capacity of the carbon. In Figure 7, the loadings with and without cooling and drying are plotted.

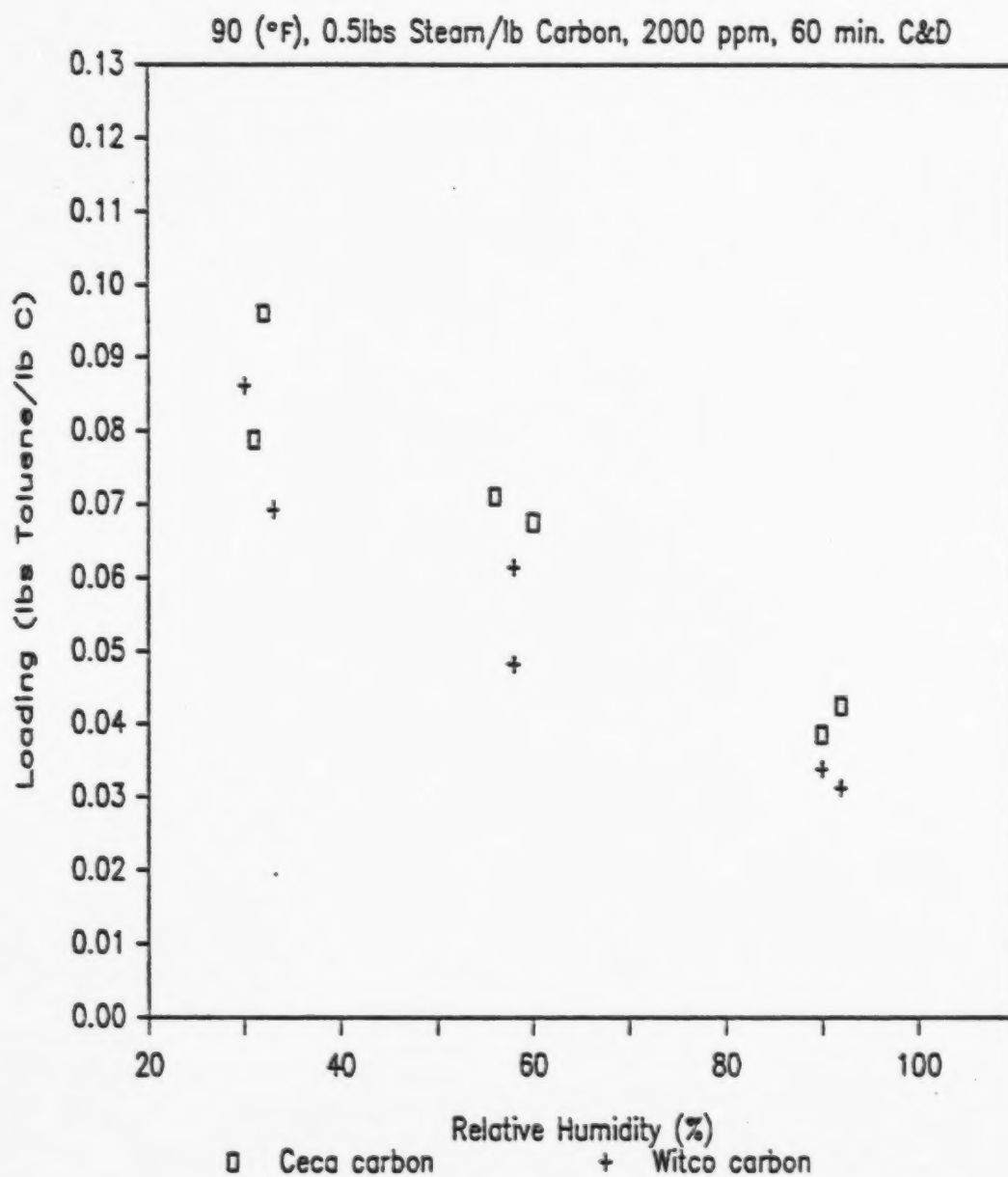


Figure 4. Effect of Relative Humidity on Loading of Toluene

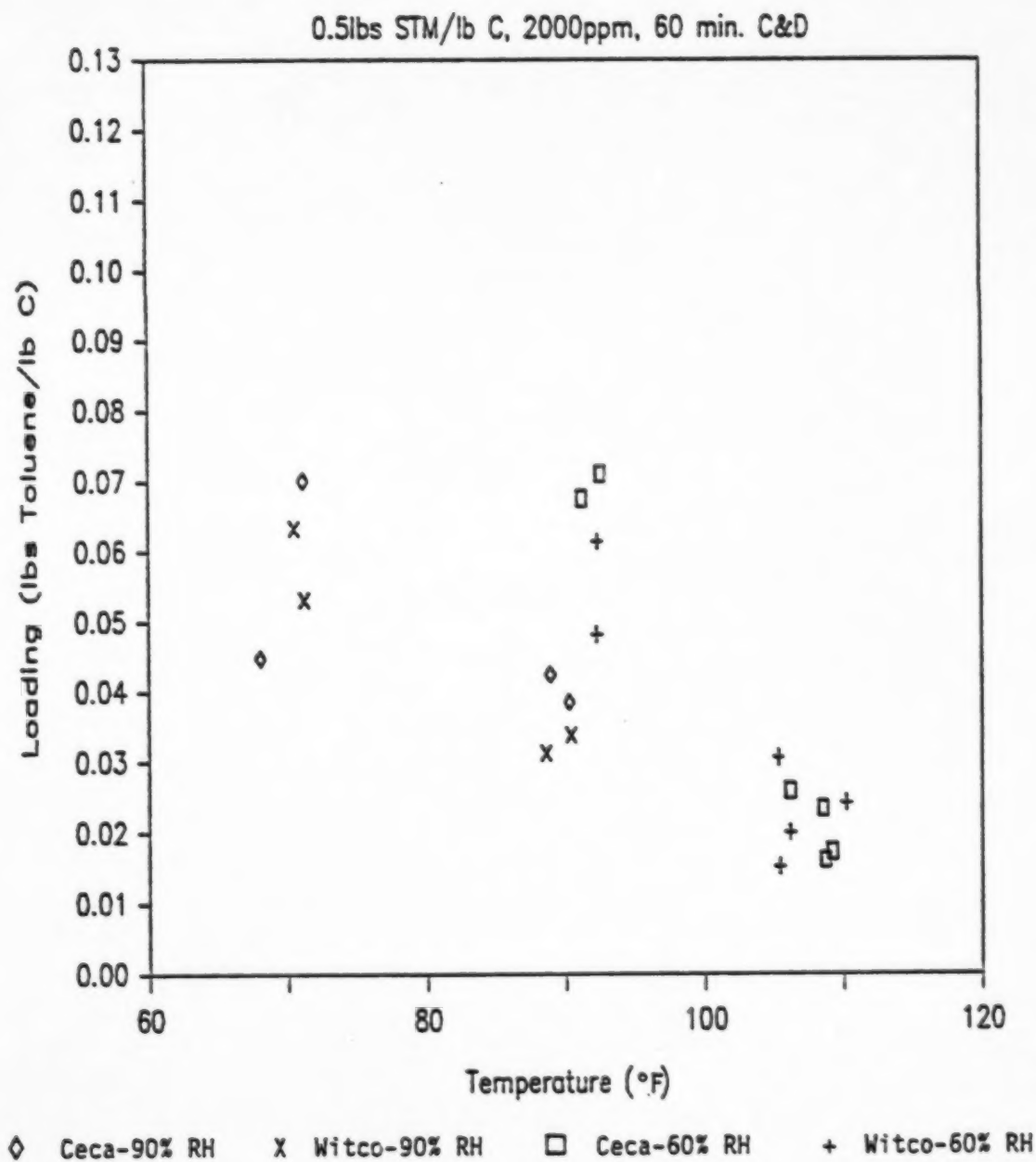


Figure 5. Effect of Temperature on Loading of Toluene

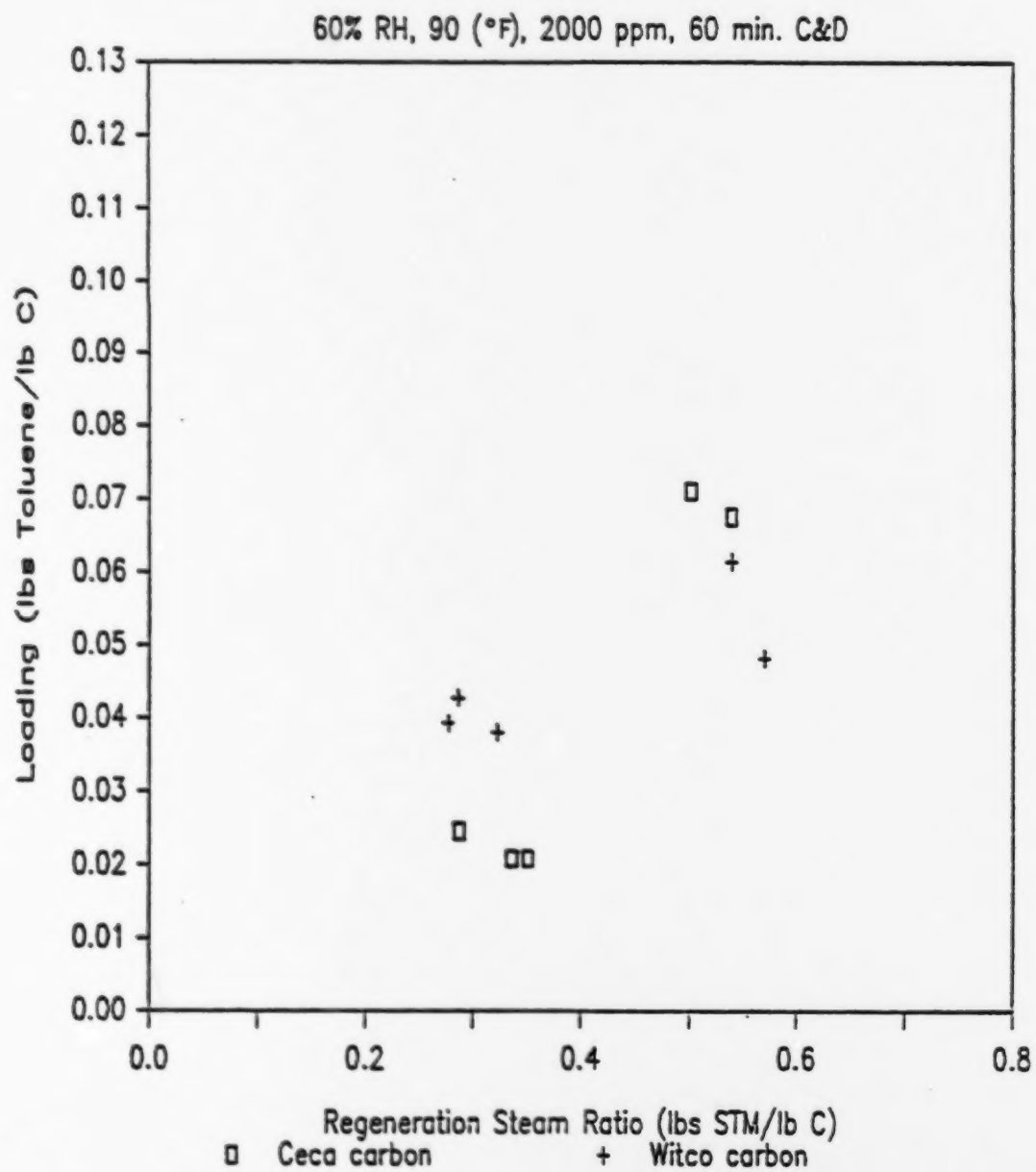


Figure 6. Effect of Regeneration Steam Ratio on Loading of Toluene

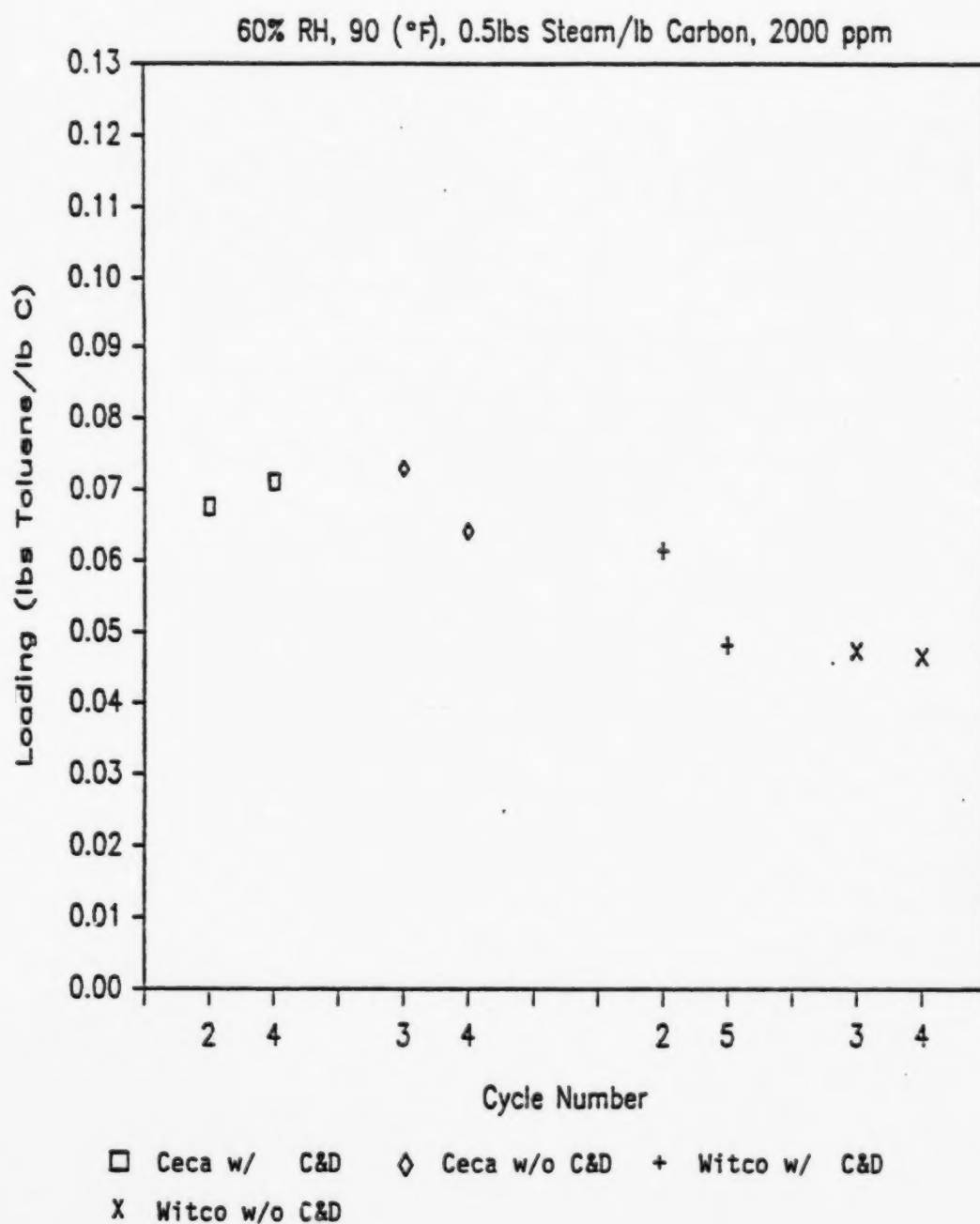


Figure 7. Effect of Cooling and Drying Step on Loading of Toluene

CONCLUSIONS

The testing performed at this site indicates the relative humidity, temperature, and the amount of steam used in regeneration all impact significantly the loading of VOC onto activated carbon. Effects of the cooling and drying cycle and the carbon type on loading were less evident.

Relative humidity and temperature are two parameters which cannot be entirely separated. From our testing it is evident that, at a constant temperature, increasing the relative humidity of the solvent-laden air from 30 to 90 percent will cut the loading by about 50 percent. Similarly, increasing the temperature by 20 degrees Fahrenheit at a constant relative humidity will also reduce the loading by about 50 percent. While these effects are evident, it is not clear whether the humidity ratio (mass of water vapor per unit mass of dry air) is a variable to be considered. When relative humidity is held constant and temperature is increased, the humidity ratio also increases.

Reducing the regeneration steam ratio significantly affected the loading for both types of activated carbons. The loading for the Witco brand was reduced by about 25 percent while the loading for the Ceca brand was reduced by about 50 percent. While the effects are significant, the absolute values are compromised by the steam control problems encountered at this site.

While lack of a cooling and drying cycle does not seem to significantly affect the loading capacity of the carbon, the effect on overall operating efficiency is more pronounced. The shape of the breakthrough curve of an adsorption cycle which directly follows a regeneration cycle has a great deal more of a leading edge than a normal curve. During this period very little adsorption takes place. Essentially, the initial few minutes of the adsorption cycle is forced to take the place of the cooling and drying cycle.

Based on the testing performed at this site, the host company has decided to implement modifications to their present system to improve the recovery of toluene from their process.